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(54) Title: A METHOD FOR THE MANUFACTURE OF CHEMITHERMOMECHANICAL PULP

(57) Abstract

A method for producing chemithermomechanical pulp from lignocellulosic material, by preheating and refining the material at an elevated temperature of 160-180 °C, subsequent to pre-impregnating the chips with an impregnating liquid that contains borohydride, at least one compound chosen from the group sodium sulphite, sodium hydrosulphite and sodium disulphite, and optionally one or more sequestering agents. The impregnating liquid preferably has a neutral, near neutral or slightly acid pH. The inventive method enables pulp discoloration to be avoided and/or a reduction in pulp brightness to be counteracted in the preheating and refining processes. The invention also relates to an impregnating liquid for impregnating lignocellulosic material in the production of chemithermomechanical pulps.

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# A METHOD FOR THE MANUFACTURE OF CHEMITHERMOMECHANICAL PULP

## The Field and Background of the Invention

The present invention relates to a method for the manufacture of chemithermomechanical pulp (hereinafter referred to as CTMP-pulps), and particularly to a method for avoiding pulp discolouration and/or for counteracting a reduction in pulp brightness in the manufacture of a CTMP-pulp at elevated temperature, and also to an impregnating liquid used when practicing the method.

Paper pulp produced from lignocellulosic material is manufactured by subjecting raw material in the form of wood chips to a mechanical, a chemical and a thermal treatment process, these processes either being effected simultaneously or separately. CTMP-pulp is manufactured conventionally by preheating and refining the wood chips at a temperature of about 100-130°C. It has now been found that energy consumption can be reduced to about half the energy that is consumed when refining CTMP-pulps conventionally at 130°C, by preheating and refining the pulps at an elevated temperature of 160-180°C. A pulp refining process which is so lean in energy is highly desirable and has also arrived at the right time, so to speak, and, furthermore, will probably become a necessity in the future, since energy prices are expected to increase considerably.

It has earlier been considered impossible to manufacture bright mechanical pulp subsequent to preheating and refining at temperatures higher than about 130°C, since pulp brightness is impaired at higher temperatures. It has also been considered impossible to bleach pulps

which have been refined at higher temperatures in subsequent bleaching stages at reasonable costs and at reasonable chemical consumptions. Furthermore, when refining at said higher temperatures, the fibres are liable to become covered with lignin, which may result in impaired strength and absorption properties. These latter properties are particularly important in the case of absorbent pulps. Consequently, refinement of the pulp has normally been effected at a temperature of at most 140°C and often at a temperature beneath 130°C. In the manufacture of highly absorbent pulps intended for the production of fluff and tissue, a high freeness and low shive content is desired, i.e. the pulp freeness index will preferably lie above 600 ml CSF, preferably above 700 ml CSF, and the shive content will not exceed 10%, preferably will lie beneath 6%.

## Disclosure of the Invention and the Object Thereof

The inventive method enables pulp to be refined at an elevated temperature - with the intention of saving energy - without impairing pulp brightness. The pulp produced can also be bleached with peroxide in a subsequent bleaching stage. The inventive method is characterized by pre-impregnating the wood chips with an impregnating liquid that contains borohydride, at least one compound chosen from the group sodium sulphite, sodium hydrosulphate and sodium disulphite, and optionally one or more sequestering agents, wherein the impregnating liquid has a neutral, a near neutral or a slightly acid pH-value, and by preheating the impregnated chips to a temperature of 160-180°C and carrying out the refining process while maintaining said temperature.

The borohydride is suitably added in the form of an alkaline aqueous solution, for instance the borohydride

solution retailed commercially under the trade name BOROL® and which contains 12 percent by weight $NaBH_4$. The borohydride is added in the form of said aqueous solution in an amount of about 5-15 kg BOROL® per tonne of chips, preferably about 10 kg per tonne of chips. Thus, the amount of active borohydride added should correspond to about 0.6-1.8 kg $NaBH_4$ per tonne of chips, preferably about 1.2 kg per tonne of chips.

The sulphite-containing compound consists of sodium sulphite, sodium hydrosulphite, sodium disulphite, or a mixture of two or more of these compounds. Calculated as the amount of sodium sulphite, $Na_2SO_3$, added to the system the total sulphite charge is roughly 20-40 kg per tonne of chips, preferably about 30 kg per tonne of chips, or corresponding stoichiometric quantities of $SO_2$, $HSO_3^-$ and/or $S_2O_5^{2-}$. In order to achieve maximum pulp brightness, the impregnating liquid should be given a neutral pH, a near neutral pH or a slightly acid pH, i.e. the pH of the liquid should lie between about 5 and 7, preferably from 5.5-6.5. This pH-value can be obtained with the aid of an acid, for instance sulphuric acid, $H_2SO_4$, or with the aid of sulphur dioxide, $SO_2$, which is often available in the pulp mill. When $SO_2$ is used to acidify the impregnating liquid, or when $HSO_3^-$ or $S_2O_5^{2-}$ is used instead of $SO_3^{2-}$, the total sulphite charge must, however, be adapted so as to lie within the aforesaid pH-range. The manner in which this sulphite charge and the recommended pH-value are achieved is, however, unimportant to the invention and may be carried out in any appropriate manner when practicing the invention.

One or more sequestering agents are also preferably added to the impregnating liquid, in order to bind in the pulp any metal ions that may be which are liable to

cause discolouration. A suitable sequestering agent in this regard is the sodium salt of diethylenetriamine pentaacetic acid (DTPA), or ethylenediamine tetraacetic acid (EDTA), said sequestering agent being added in an amount of about 2-4 kg per tonne of chips.

The invention also relates to an impregnating liquid intended for the impregnation of lignocellulosic material in the manufacture of chemithermomechanical pulp. The impregnating liquid includes borohydride, at least one compound selected from the group sodium sulphite, sodium hydrosulphite and sodium disulphite, and optionally one or more sequestering agents. The weight ratio between the active components borohydride and total sulphite concentration of the liquid is between 20:80 and 30:70, preferably about 25:75. The pH of the impregnating liquid is adjusted to a neutral value, a near neutral value or a weakly acid value, in other words the pH-value of the liquid will preferably lie between 5 and 7, more preferably between 5.5-6.5.

When producing wood pulp in accordance with the present invention, wood chips are impregnated with an impregnating liquid that contains borohydride and at least one compound selected from the group sodium sulphite, sodium hydrosulphite and sodium disulphite, and optionally one or more sequestering agents. The impregnated chips are then preheated over a period of from 1-3 minutes, preferably over a period of about 1 minute, at a temperature of 160-180°C, preferably about 165-170°C, whereafter the wood chips are defibred/refined to pulp form while essentially maintaining pressure and temperature from the preheating stage. The pulp is suitably washed under pressure and high temperature, preferably without the ingress of air, in immediate connection with the refining process, the pulp being dewatered to a

consistency of 25-40%, and then bleached with peroxide. The pulp can be rewashed after the bleaching stage, if necessary.

The pulp is refined to a freeness value in excess of 700 ml CSF, at an energy consumption of about 500 kWh/ tonne.

The aforedescribed method enables a pulp of unbleached brightness to be produced by high temperature refinement prior to the peroxide bleaching stage, this brightness being comparable to the brightness of conventionally-produced CTMP-pulps. When the pulp produced in accordance with the invention is subjected to a following peroxide bleaching stage there is also obtained an equivalent bleaching response and a maximum brightness of the same order of magnitude as that achieved with pulps produced conventionally at lower temperatures.

Working Example

Example 1. (High temperature refinement with borohydride).

Spruce chips were impregnated with impregnating liquids containing 30 kg sodium sulphite, 10 kg BOROL® (12% aqueous solution of sodium borohydride) and 3 kg DTPA per tonne of chips. The impregnating liquid had a pH = 11, or alternatively was neutralized or acidified with $H_2SO_4$ to pH = 8 and pH = 6 respectively. The chips were preheated at a temperature of 165°C for a period of 1 minute. The chips were then refined while maintaining the aforesaid temperature.

Subsequent to this refinement, the pulp was washed and dewatered in immediate connection with the refining

process under pressure and at high temperature, about 150-180°C, without the ingress of air into the pulp. Reference is made to the following Table 1 with regard to other process conditions.

The brightness of the unbleached pulps produced was measured in accordance with SCAN-C 11 at a wavelength of 457 nm.

The produced pulps were then bleached in a conventional manner, using 40 kg of peroxide per tonne of pulp. The brightness of the pulps was measured in the same manner as that used to measure the brightness of the unbleached pulps. The brightness values measured are set forth in Table 1 below.

Example 2. (High temperature refinement without borohydride).

Spruce chips were impregnated with an impregnating liquid having a pH = 9.5 and containing 30 kg $Na_2SO_3$ and 3 kg of DTPA per tonne of chips. The chips were heated to 165°C over a period of 1 minute and were treated, in other respects, in accordance with Example 1. The brightness of the unbleached pulp and the brightness of the pulp bleached with peroxide were measured in accordance with SCAN-C 11 and the results are set forth in Table 1 below.

Example 3. (Conventional refinement).

Spruce chips were impregnated with impregnating liquid similar to that used in Example 2. The chips were preheated to 130°C over a period of 3 minutes and refined while maintaining this temperature. Subsequent to refinement, the pulp was washed and dewatered in a

conventional manner at about 130°C, without the ingress of air into the pulp.

The brightness of the unbleached pulp and the brightness of the pulp bleached with peroxide were measured and the results obtained are set forth in Table 1 below.

The results obtained with the three methods described in Examples 1-3 above and the process conditions applicable to said methods are collected in Table 1.

Table 1

| | Ex. 1 | | | Ex. 2 | Ex. 3 |
|---|---|---|---|---|---|
| Process conditions: | | | | | |
| Preheat temp. °C | 165 | | | 165 | 130 |
| Time min. | 1 | | | 1 | 1 |
| Energy consumption kWh/t | 550 | | | 600 | 900 |
| Chip impregnation: | | | | | |
| $Na_2SO_3$ kg/t | 30 | | | 30 | 30 |
| BOROL® (12 percent by weight $NaBH_4$) kg/t | 10 | | | - | - |
| DTPA kg/t | 3 | | | 3 | 3 |
| pH-value | 6 | 8 | 11 | 9.5 | 9.5 |
| Result: | | | | | |
| Freeness CSF ml | 740 | | | 740 | 730 |
| Unbleached brightness after refinement ISO % | 59 | 57.5 | 55 | 54 | 57 |
| Bleached brightness after peroxide bleaching ($H_2O_2$, 40 kg/t) ISO % | 78.5 | 77 | 75 | 73 | 78 |

It is evident from the results obtained with the brightness measurements made on the produced pulps that the high temperature refinement carried out with the intention of reducing the amount of energy consumed by the refinement process can be achieved while maintaining the brightness of the pulps produced provided that the chips are pre-impregnated with an impregnating liquid in accordance with the invention. The measured brightness values also show that the pH of the impregnating liquor has an effect on the result.

CLAIMS

1. A method for producing chemithermomechanical pulp from lignocellulosic material, by preheating and refining at an elevated temperature chips pre-impregnated with a liquid that contains borohydride and at least one compound chosen from the group sodium sulphite, sodium hydrosulphite and sodium disulphite, and optionally one or more sequestering agents, c h a r a c t e r i z e d in that said impregnating liquid having a slightly acid pH-value of between 5 and 7; and the impregnated chips are preheated to a temperature of between 160-180°C and the refining of the chips is carried out while generally maintaining said temperature.

2. A method according to Claim 1, c h a r a c - t e r i z e d by impregnating each tonne of chips with 0.6-1.8 kg of sodium borohydride and 20-40 kg of sodium sulphite or a corresponding stoichiometric quantity of sodium hydrosulphite and/or sodium disulphite or a mixture of at least two of the aforesaid compounds.

3. A method according to any one of Claims 1-2, c h a r a c t e r i z e d in that the impregnating liquid is acidified to a pH of between about 5.5 and 6.5, optionally after adding acid.

4. A method according to any one of Claims 1-3, c h a r a c t e r i z e d by carrying out the preheating process at a temperature between 160-180°C over a time period of about 1-3 minutes.

5. A method according to any one of Claims 1-4, c h a r a c t e r i z e d by impregnating the chips with an aqueous solution containing about 1.2 kg boro-hydride, about 30 kg sodium sulphite and about 3 kg

DTPA per tonne of chips, and then preheating the chips to about 165-170°C over a period of 1 minute and refining said chips while generally maintaining said temperature.

6. A method according to any one of the preceding Claims, characterized by bleaching the produced pulp with peroxide in a conventional manner.

7. Impregnating liquid containing borohydride and at least one compound chosen from the group sodium sulphite, sodium hydrosulphite and sodium disulphite, and optionally one or more sequestering agents for the impregnation of lignocellulosic material in the production of chemithermomechanical pulp, characterized in that the impregnating liquid has a slightly acid pH-value of between 5 and 7.

8. An impregnating liquid according to Claim 7, characterized in that the liquid has a pH-value of between about 5 and 7, preferably between 5.5 and 6.5.

9. An impregnating liquid according to any one of Claims 7-8, characterized in that the active components borohydride and total sulphite are present in the liquid in an amount ratio of between 20:80 and 30:70.

# INTERNATIONAL SEARCH REPORT

International Application No PCT/SE 92/00348

## I. CLASSIFICATION OF SUBJECT MATTER (if several classification symbols apply, indicate all)[6]

According to International Patent Classification (IPC) or to both National Classification and IPC

IPC5: D 21 B 1/02

## II. FIELDS SEARCHED

Minimum Documentation Searched[7]

| Classification System | Classification Symbols |
|---|---|
| IPC5 | D 21 B |

Documentation Searched other than Minimum Documentation to the Extent that such Documents are included in Fields Searched[8]

SE,DK,FI,NO classes as above

## III. DOCUMENTS CONSIDERED TO BE RELEVANT[9]

| Category * | Citation of Document,[11] with indication, where appropriate, of the relevant passages[12] | Relevant to Claim No.[13] |
|---|---|---|
| X | EP, A1, 0293309 (ATOCHEM) 30 November 1988, see the whole document | 1,2,6,7, 9 |
| A | -- | 3,4,5,8 |
| A | TAPPI PROCEEDINGS, Pulping Conference, 1988, Oct. 30 - Nov. 2, Book 2, Atlanta: Tappi Press, 1988, New Orleans, Devic Michel et al: "Improved brightness and strength by sulfonation in presence of a reducing agent", page 491 - page 495 | 1-9 |
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## IV. CERTIFICATION

| Date of the Actual Completion of the International Search | Date of Mailing of this International Search Report |
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| 2nd September 1992 | 1992 -09- 07 |
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# ANNEX TO THE INTERNATIONAL SEARCH REPORT ON INTERNATIONAL PATENT APPLICATION NO.PCT/SE 92/00348

This annex lists the patent family members relating to the patent documents cited in the above-mentioned international search report. The members are as contained in the Swedish Patent Office EDP file on 31/07/92
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| Patent document cited in search report | Publication date | Patent family member(s) | | Publication date |
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